Effect of Cooking Conditions on Selected Properties of Biodiesel Produced from Palm-Based Waste Cooking Oils

Vladimir Plata [1,*], **Deyanira Ferreira-Beltrán** [2] **and Paola Gauthier-Maradei** [2]

[1] Grupo de Investigación en Ciencias Básicas y Aplicadas, Departamento de Ciencias Básicas, Universidad Santo Tomás Seccional Bucaramanga, Bucaramanga 68001, Colombia

[2] Grupo de Investigación en Tecnologías de Valorización de Residuos y Fuentes Agrícolas e Industriales para la Sustentabilidad Energética, Escuela de Ingeniería Química, Universidad Industrial de Santander, Bucaramanga 68001, Colombia; deyanira2188239@correo.uis.edu.co (D.F.-B.); mapaomar@uis.edu.co (P.G.-M.)

[*] Correspondence: vladimir.plata@ustabuca.edu.co; Tel.: +57-7-6985858

Abstract: Cooking conditions affect oil properties and consequently, the quality of the derived biodiesel. Nevertheless, little information regarding the impact of the cooking process on biodiesel properties is currently available, especially for palm biodiesel. Therefore, this study examined the effect of cooking temperature, time of use, and length of reuse, on selected properties of biodiesel produced from palm-based waste cooking oils (WCO). Several WCO collected from restaurants belonging to four categories, namely fried chicken restaurants, fast food restaurants, snack producers, and typical restaurants, were subjected to base-catalyzed transesterification. The biodiesel yield was calculated, and the produced biodiesel was characterized as to its kinematic viscosity, calorific value, and cetane number. As a result, palm-based WCO performed better than other WCO in terms of biodiesel yield regardless of the conditions that they experienced, achieving almost 95% in some cases. The yield decreased as the cooking temperature and length of reuse moved upward, whereas the kinematic viscosity was sensitive only to the length of reuse, rising with increasing reuse. Non-compliance with biodiesel standards and technical requirements was observed in a few cases. The calorific value did not significantly change unless the cooking conditions were severe. The cetane number dropped as use and reuse decreased, remaining better compared to petrodiesel (70.2 ± 3.2 on average). Typical restaurants would generate the most suitable WCO to produce biodiesel, i.e., yield: $93.1 \pm 0.2\%$; kinematic viscosity: 5.0 ± 0.3 mm^2/s; calorific value: 39.9 ± 0.1 MJ/kg; density: 919 ± 9 kg/m^3; and cetane number: 67.4 on average. This is consistent with the less severe cooking conditions employed at these restaurants.

Keywords: cooking conditions; frying oils; physical properties; transesterification; biodiesel yield

1. Introduction

In recent years, biodiesel has attracted global attention due to its potential for replacing conventional diesel fuel [1]. In addition, it offers desirable characteristics, such as being renewable, biodegradable, and non-toxic [2]. Biodiesel is produced mainly through a transesterification process, which involves the chemical reaction of lipid feedstock (containing mostly triglycerides) with short-chain alcohol to yield fatty acid alkyl esters (biodiesel) and glycerol as byproducts (Figure 1) [3]. A strong base catalyst, e.g., NaOH, KOH, CH$_3$ONa, and CH$_3$OK, improves the transesterification reaction rate [4]. Edible oils, including soybean, rapeseed, and palm oil, are the principal biodiesel feedstocks. However, the high cost of these oils represents the main barrier to the commercialization of biodiesel [5].

Using waste cooking oils (WCO) as a biodiesel feedstock not only causes a significant reduction in production costs but also eliminates competition within the food industry; it also offers an acceptable way of disposing of such waste [3]. Nevertheless, a drawback of producing biodiesel from frying oils is that this material contains several impurities that

may lead to undesirable side reactions, such as saponification and hydrolysis, decreasing the biodiesel yield and affecting its quality [6]. Figure 2 depicts a simplified process diagram.
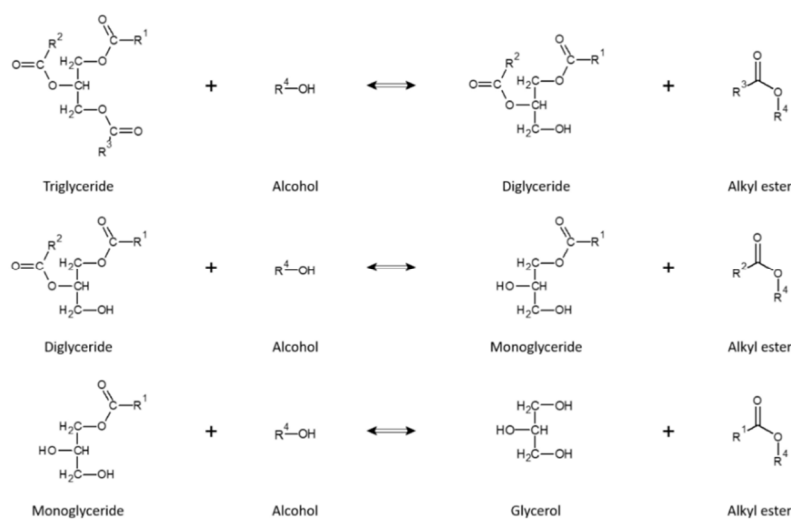

Figure 1. Transesterification reaction scheme. R^1, R^2, and R^3 represent the triglyceride fatty acid chains. R^4 represents the alcohol chain.

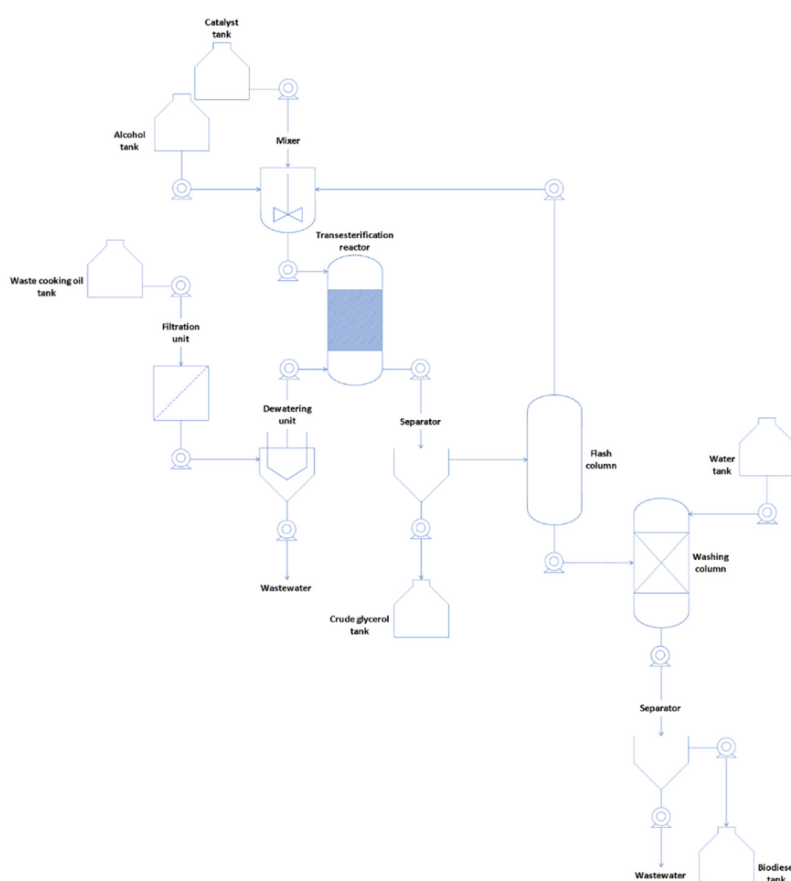**Figure 2.** Simplified process diagram for waste cooking oil transesterification.

Conventionally, biodiesel production from WCO comprises a series of upstream and downstream steps involving separation processes, apart from the transesterification reaction [7]. First, suspended solids are removed by filtration, and moisture is eliminated by heating above 100 °C. Then, triglycerides are converted into fatty acid alkyl esters

upon reacting pretreated WCO and an alcoholic solution of the base catalyst. Afterward, the reaction products are separated, employing a gravity settler. Finally, the biodiesel is purified, utilizing a flash column for alcohol removal and a wash column to eliminate the catalyst and soap. Cooking conditions, namely the cooking temperature, time of use, and length of reuse, affect the oils properties, and consequently, the quality of biodiesel derived thereof [8]. Several studies have examined the changes in oils exposed to varying conditions [9]. However, little information regarding the impact of the cooking process on biodiesel properties is currently available to the best of our knowledge. So far, the effect of the cooking temperature and time on selected properties of biodiesel produced from canola-, sunflower-, and corn-based WCO has been evaluated [10]. Nevertheless, the impact of the length of reuse, which is a critical cooking condition [9], has not been investigated nor has the effect on biodiesel yield. Therefore, this study examined the effect of cooking conditions on the yield and selected properties of biodiesel produced from palm-based WCO. Since the variability in the WCO properties among restaurants may concern their use as a biodiesel feedstock [8], determining which type of restaurant would generate the most suitable WCO is a related objective.

2. Materials and Methods

2.1. Materials

WCO were collected from 8 restaurants in Bucaramanga, Colombia. The restaurants belonged to four categories: Fried chicken restaurants (FCR), fast food restaurants (FFR), snack producers (SP), and typical restaurants (TR). Data on the cooking conditions employed at each restaurant was obtained using a short survey completed by persons in charge of the restaurants.

2.2. WCO Conditioning

The WCO were conditioned by filtration through a cloth sieve (47-mm diameter) under vacuum for 10 min followed by centrifugation at 5800 rpm for 20 min and filtration through Whatman No. 2 filter paper (47-mm diameter) under vacuum for 10 min. The oils were then stored in dark bottles at 25 °C under a dry nitrogen atmosphere.

2.3. WCO Characterization

The WCO were characterized as to their acid value, peroxide value, and viscosity at 40 °C following the AOAC 940.28 [11], AOAC 965.33 [12], and ASTM D2196 Standard Test Method [13], respectively. Each analysis was conducted in duplicate.

2.4. Biodiesel Production and Characterization

The WCO were transesterified using a sealed 1-L glass reactor equipped with a methanol/KOH mixture container, a reflux condenser, a heating bath, a temperature controller, and a magnetic stirrer (Figure 3). First, 200 g of oil were placed in the reactor, stirred at 400 rpm, and heated at 60 °C. Then, a mixture of methanol (44 g) and KOH (2 g) was added to the oil, and the resulting mixture reacted for 60 min. As a result, the methanol/oil molar ratio was 6:1, and the KOH/oil mass ratio was 0.01. Afterward, the reaction mixture was transferred to a separatory funnel and left to settle for 12 h. Then, the glycerol layer was removed, and the biodiesel layer was washed several times with distilled water at 50 °C (water/biodiesel 1:1 volume ratio) until reaching a neutral pH. After washing, the biodiesel was heated at 110 °C for 20 min to remove excess methanol and water and stored in dark bottles at 25 °C under a dry nitrogen atmosphere.
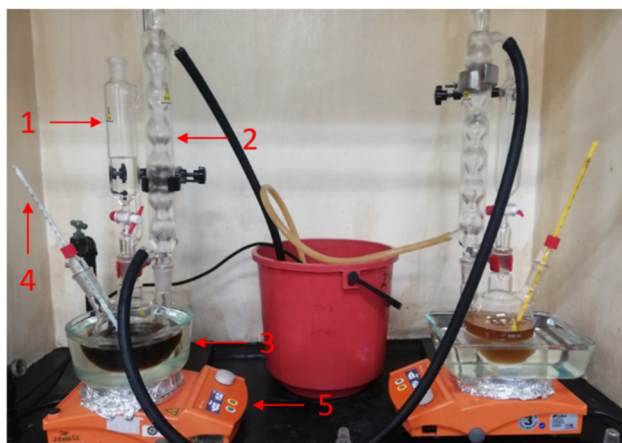
Figure 3. Photograph view of the reactor utilized for biodiesel production, including (1) a methanol/KOH mixture container, (2) a reflux condenser, (3) a heating bath, (4) a temperature controller, and (5) a magnetic stirrer.

The biodiesel yield was calculated according to Equation (1):

$$Biodiesel\ yield\ (\%) = \frac{Mass\ of\ biodiesel\ produced}{Initial\ mass\ of\ WCO} \times 100. \tag{1}$$

The biodiesel fatty acid profile was determined by Laboratorio de Cromatografía y Espectrometría de Masas (CROM-MASS) at Universidad Industrial de Santander (Bucaramanga, Colombia) in accordance with the ISO 5508 and 5509 Standards [14,15]. Viscosity at 40 °C was determined following the ASTM D2196 Standard Test Method [13]. Calorific value was measured using a PARR 6200 calorimeter bomb. Density at 40 °C was determined using a calibrated pycnometer. The cetane number was predicted using Equation (2):

$$Cetane\ number = \sum \%ME \cdot CN_{ME} \tag{2}$$

where %ME and CN_{ME} are the mass% and cetane number of each methyl esters, respectively [16]. Each analysis was conducted in duplicate. To better understand the cooking conditions' impact on the cetane number, the unsaturation degree (UD) was calculated using Equation (3) and the biodiesel fatty acid profile:

$$UD = (1 \cdot \%MU + 2 \cdot \%DU + 3 \cdot \%TU)/100. \tag{3}$$

where %MU is the total mass% of monounsaturated fatty acid, %DU is the total mass% of diunsaturated fatty acid, and %TU is the total mass% of triunsaturated fatty acid [16].

2.5. Statistical Analysis

Data were analyzed by ANOVA followed by the Fisher's Least Significant Difference (LSD) test for multiple comparisons using Statgraphics Centurion software (free trial version, StatPoint Technologies, Inc., Warrenton, VA, USA). A *p*-value of <0.05 was considered statistically significant.

3. Results and Discussion

3.1. Fatty Acid Composition

Palmitic acid (C16:0) and oleic acid (C18:1) were the primary fatty acids of the WCO (Table 1). Stearic acid (C18:0), linoleic acid (C18:2), and linolenic acid (C18:3) were also present, although at lower levels. This is characteristic of palm-based oils [17]. Some oils, especially TR2, seemed to have a comparatively low palmitic acid content. However, the fatty acids profile corresponded to saturated oils (41.44% of saturates on average).

Table 1. Fatty acid composition of representative palm-based waste cooking oils (WCO). Relative standard deviation was within 2% of average (n = 2). FCR denotes fried chicken restaurants; FFR denotes fast food restaurants; SP denotes snacks producers; and TR denotes traditional restaurants.

WCO	C16:0	C16:1	C18:0	C18:1	C18:2	C18:3	C20:0	∑SFA [a]	∑UFA [b]
FCR1	40.36	3.92	2.78	51.68	0.65	0.31	0.30	43.44	56.56
FCR2	34.62	2.30	1.79	60.31	0.50	0.22	0.26	36.67	63.33
FFR1	43.47	1.15	2.55	51.53	0.63	0.22	0.44	46.47	53.53
FFR2	33.98	0.98	2.44	59.87	2.08	0.22	0.42	36.84	63.16
SP1	48.47	1.33	2.49	46.69	0.14	0.47	0.40	51.37	48.63
SP2	50.97	1.82	2.60	43.02	0.20	0.92	0.46	54.04	45.96
TR1	42.34	2.40	3.03	50.87	0.64	0.33	0.39	45.76	54.24
TR2	13.93	0.52	2.57	78.43	3.75	0.40	0.41	16.91	83.09
Average								41.44	58.56

[a] ∑SFA = C16:0 + C18:0 + C20:0; [b] ∑UFA = C16:1 + C18:1 + C18:2 + C18:3.

3.2. Biodiesel Yield

According to ANOVA and LSD multiple range test results, biodiesel yield means were grouped into four homogeneous groups identified by "a" to "d" (Figure 4), each of which was significantly different from the rest at a 95% level of confidence (p-value < 0.0001). On the other hand, no significant differences existed among TR1, TR2, FCR2, FFR2, and FFR1. Hence, FFR1 was identified by combining "a" and "b."
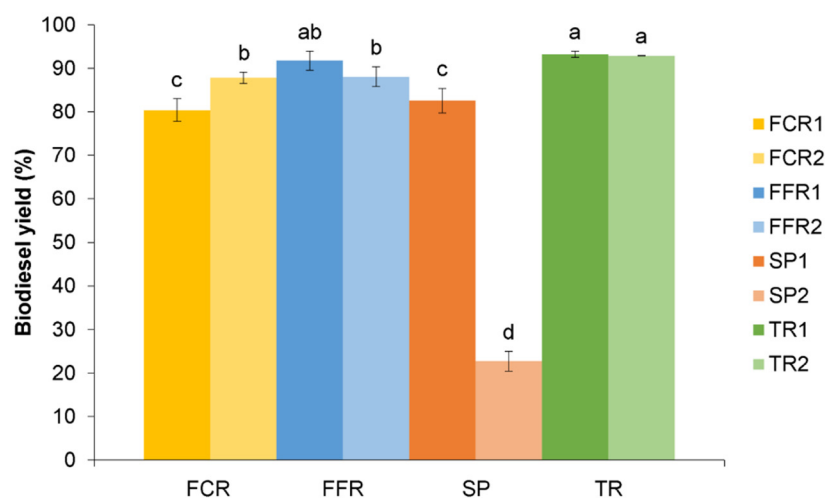

Figure 4. Biodiesel yield provided by representative palm-based waste cooking oils. Means with different letters are significantly different by LSD (Least Significant Difference) multiple range test (p-value < 0.05). Error bars show standard deviation. For details on abbreviations, refer to Table 1.

As shown in Figure 4, TR1 and TR2 (group "a") provided the highest biodiesel yield, followed by FCR2 and FFR2 (group "b"), FCR1 and SP1 (group "c"), and SP2 (group "d"). Except for groups "c" and "d," the yield was >88%, and for group "a," it was close to 95% (Figure 4). These results overcome those reported for other WCO. For example, Wyse-Mason and Beckles transesterified several soybean-based WCO using a base catalyst and achieved a yield of 82% at best [7].

The low biodiesel yield given by FCR1 and SP2 is consistent with their high acid value (Table 2). During base-catalyzed transesterification, the free fatty acids in oils react with the catalyst to form soaps. As a consequence, the oil conversion into biodiesel decreases [18]. Nevertheless, the yield provided by FCR1 and SP2 confirmed that palm-based WCO performed better as biodiesel feedstock. Wyse-Mason and Beckles attained <6% with soybean-based WCO subjected to similar cooking conditions [7].

Table 2. Selected properties of representative palm-based waste cooking oils. Relative standard deviation was within 3% of the average. For details on abbreviations, refer to Table 1.

WCO	Acid Value mg KOH/g	Peroxide Value meq O_2/kg	Dynamic Viscosity cP
FCR1	5.17	6.62	42.4
FCR2	2.71	2.19	48.8
FFR1	1.31	2.84	45.8
FFR2	3.14	5.91	47.7
SP1	1.26	12.47	48.3
SP2	8.79	6.53	72.5
TR1	1.04	6.53	46.7
TR2	1.87	2.04	34.1

The high acid value of FCR1 and SP2 is attributable to the severe conditions they experienced (Table 3). FCR1 was exposed to >300 °C, whereas SP2 was subjected to >13 h of daily use. During cooking, the moisture in oils and food hydrolyzes the ester bond of triglycerides, and prolonged the contact of oils with the aqueous phase as high temperatures increases hydrolysis and the free fatty acids content [9]. Thus, FCR1 and SP2 gave poor biodiesel yields.

Table 3. Cooking conditions that representative palm-based waste cooking oils experienced. For details on abbreviations, refer to Table 1.

WCO	Cooking Temperature (°C)	Time of Use (h/Day)	Length of Reuse (Day)	Food Cooked
FCR1	>300	3–5	3–7	Chicken, fries, plantain slices
FCR2	>300	8–13	1–3	Chicken, fries
FFR1	250–300	8–13	3–7	Fries
FFR2	200–250	5–8	3–7	Fries, sausages
SP1	150–200	5–8	1–3	Potato chips
SP2	150–200	>13	3–7	Empanadas
TR1	<150	3–5	1–3	Meat, fries
TR2	<150	8–13	1–3	Meat, fries, plantain slices

FCR2 was also heated to above 300 °C. However, it provided a superior yield than FCR1 (8.5% higher), even though FCR2 was used longer. This is probably due to the difference in the length of reuse. FCR1 was reused twice as long (3–7 versus 1–3 days of consecutive reuse), raising the number of fryings. As frying moves upward, the oils become more degraded [9]. Intermittent heating results in higher deterioration because oxygen solubility increases as the oils cool down [19]. This is consistent with the higher peroxide value of FCR1. Consequently, the oil conversion appears to drop with rising reuse.

TR2 was exposed to lower temperatures than FCR2 under identical conditions of use and reuse and gave a better biodiesel yield (5.8% higher). In contrast, TR1 and TR2 showed a non-statistically significant difference, even though TR2 was subjected to more prolonged use over the same 3-day period at similar temperatures. This indicates that the cooking temperature and length of reuse have a significant effect on oil conversion, whereas the time of use does not unless it overcomes 13 h/day, as discussed above for SP2. On the other hand, the yield may not significantly vary when the cooking temperature rises slightly and, simultaneously, the length of reuse remains constant, as evidenced by FFR1 and FFR2.

FFR1 was heated to higher temperatures and used as long as SP2 over the same 7-day period. Nevertheless, it provided a marked different biodiesel yield (75.2% higher). This may seem to contradict the previous discussion, but it suggests that the type of food cooked is another factor that affects the oil conversion. As shown in Table 3, FFR1 was utilized for

cooking fries, whereas SP2 was employed to prepare empanadas. An empanada is a fried snack consisting of wheat flour pastry and filling. Both fries and wheat flour are composed of starch [20,21], which promotes oxidation [9]. However, fries have a lower starch content, causing FFR1 to be less degraded (2.21 vs. 6.53 meq O_2/kg observed for SP2).

3.3. Viscosity

According to ANOVA and LSD multiple range test results, kinematic viscosity means were grouped into three homogeneous groups identified by "a" to "c" (Figure 5), each of which was significantly different from the rest at a 95% level of confidence (*p*-value < 0.0001). On the other hand, no significant differences existed among FCR1, FFR1, FCR2, TR2, FFR2, SP1, and TR1. Hence, FFR2, SP1, and TR1 were identified by combining "b" and "c".
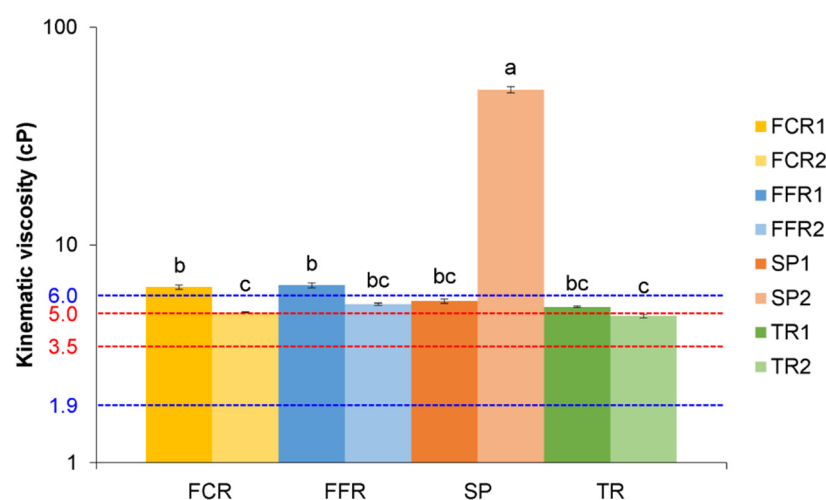

Figure 5. Kinematic viscosity of biodiesel derived from representative palm-based waste cooking oils. The blue and red dotted lines indicate the ASTM D6751 and EN 14214 limits for this property, respectively. Means having different letters are significantly different by LSD multiple range test (*p*-value < 0.05). Error bars show standard deviation. For details on abbreviations, refer to Table 1.

As shown in Figure 5, SP2 (group "a") provided the highest kinematic viscosity, followed by FCR1 and FFR1 (group "b"), and FCR2, TR2 (group "c"). With the exception of groups "a" and "b", the viscosity was within the range required by the American biodiesel standard ASTM D6751 (1.9–6.0 mm^2/s) [22]. Besides, group "c" met the stricter EN 14214 specifications (3.5–5.0 mm^2/s) [23] (Figure 5). This is desirable in biodiesel, as high viscosities demand more energy from the pump to move the fuel through the filter and lines, leading to wear on the pump [24].

No significant differences existed between FCR2 and TR2, even though FCR2 experienced higher temperatures. Similarly, TR1 did not statistically differ from TR2, even though TR2 was subjected to more extended use. By contrast, FCR1 was reused longer than FCR2 and yielded biodiesel with a higher viscosity. This evidences that biodiesel viscosity is sensitive only to the length of reuse, moving upward as reuse increases. Prolonged reuse promotes the formation of polymers of triglycerides that raise the oil viscosity, and therefore, the biodiesel viscosity [9].

3.4. Calorific Value

According to ANOVA and LSD multiple range test results, calorific value means were grouped into two homogeneous groups identified by "a" and "b" (Figure 6), each of which was significantly different from the rest at a 95% level of confidence (*p*-value < 0.0001). As shown in Figure 6, except for SP2 (group "b"), the WCO yielded biodiesel with a calorific value superior to that reported for other WCO (39.1 MJ/kg [25], 38.5 MJ/kg [26], 37.2 MJ/kg [27], 35.7 MJ/kg [3]) (Figure 6). Even so, the calorific value remained lower

compared to petrodiesel (41.2 MJ/kg) [3]. Nevertheless, the higher density of the produced biodiesel (Figure 7) compensates for its reduced energy content, so the amount of fuel injected into the combustion chamber may not require adjustment to achieve similar power outputs to petrodiesel [3]. Furthermore, all but one calorific value did not significantly change. This indicates that the cooking process does not affect the calorific value unless the conditions are severe.
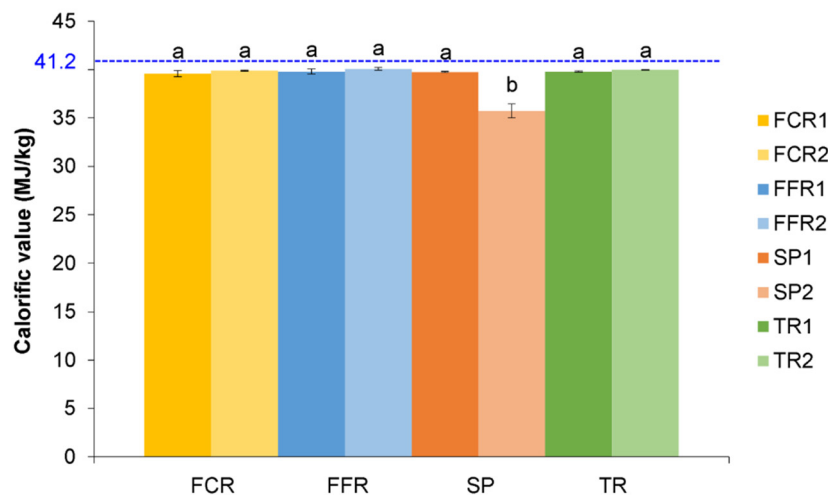

Figure 6. Calorific value of biodiesel derived from representative palm-based waste cooking oils. The blue dotted line indicates the 41.2 MJ/kg value reported for petrodiesel [3]. Means having different letters are significantly different by LSD multiple range test (*p*-value < 0.05). Error bars show standard deviation. For details on abbreviations, refer to Table 1.

Figure 7. Density of biodiesel derived from representative palm-based waste cooking oils. The blue dotted line indicates the 812 kg/m^3 value reported for petrodiesel [28]. Means having different letters are significantly different by LSD multiple range test (*p*-value < 0.05). Error bars show standard deviation. For details on abbreviations, refer to Table 1.
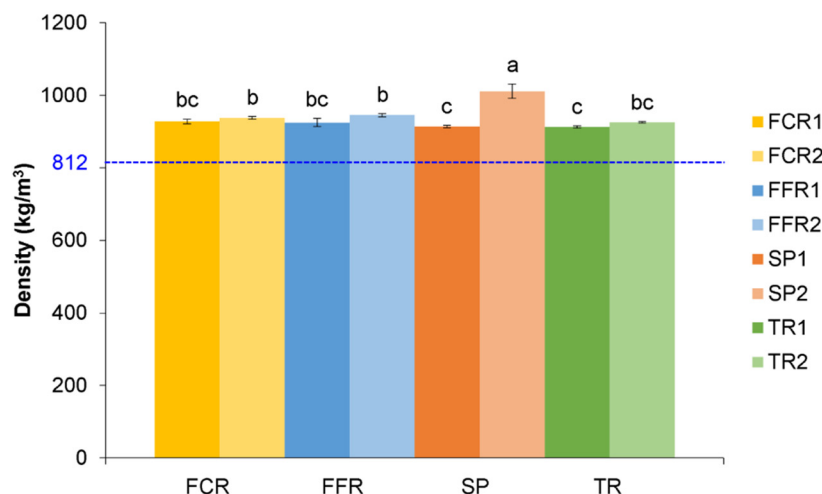
3.5. Cetane Number

The cetane number of petrodiesel is generally estimated using the cetane index [29]. In contrast, the cetane number of biodiesel is predicted using correlations based on its fatty acid profile [30]. In addition, biodiesel's ignition behavior is typically better compared to petrodiesel [31]. Accordingly, the WCO all yielded biodiesel with a superior cetane number (70.2 ± 3.2 on average versus 45 observed for petrodiesel [32]) (Figure 8).
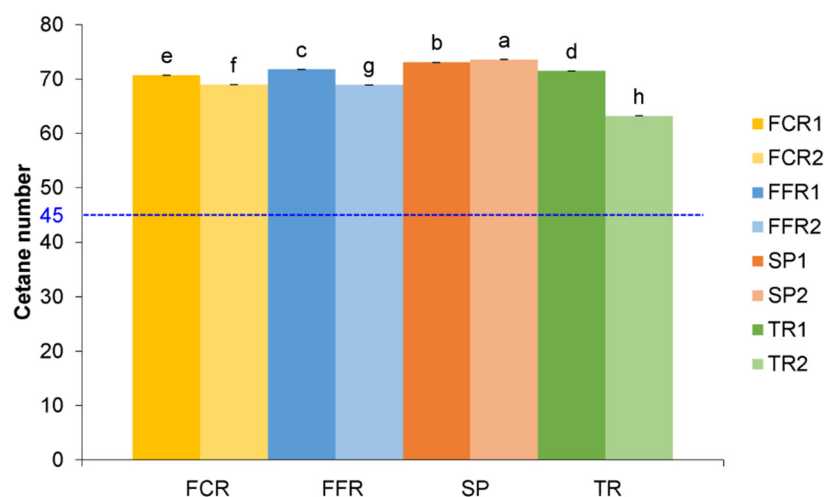
Figure 8. Cetane number of biodiesel derived from representative palm-based waste cooking oils. The blue dotted line indicates a value of 45 reported for petrodiesel [32]. Means having different letters are significantly different by LSD multiple range test (*p*-value < 0.05). Error bars show standard deviation. For details on abbreviations, refer to Table 1.

According to ANOVA and LSD multiple range test results, cetane number means were grouped into eight homogeneous groups identified by "a" to "h" (Figure 8), each of which was significantly different from the rest at a 95% level of confidence (*p*-value < 0.0001). In agreement with the literature [16], the cetane number moved upward as UD decreased (Figure 9), the highest cetane number corresponding to SP2. This is attributable to the severe conditions it experienced. During cooking, hydration of unsaturated fatty acids may occur [33], dropping the UD value. SP2 was subjected to extended use and reuse and attained the lowest UD. In consequence, the cetane number increases with rising use and reuse.
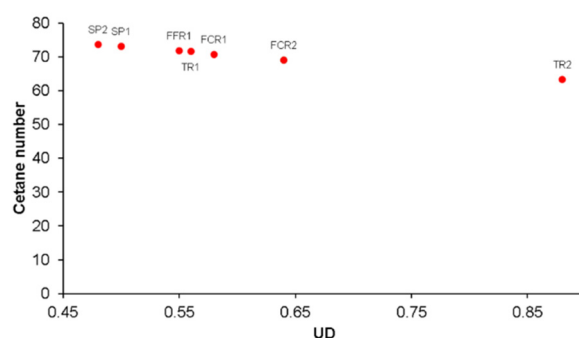

Figure 9. Relationship between the cetane number and unsaturation degree of biodiesel derived from representative palm-based waste cooking oils. For details on abbreviations, refer to Table 1.

3.6. Effect of Type of Restaurant

The properties examined demonstrated the quality of most of the produced biodiesel. However, because of the interest in determining which type of restaurant would generate the most suitable WCO to produce biodiesel, the results obtained for fried chicken restaurants, fast food restaurants, and traditional restaurants were analyzed by categories. Snacks producers were not considered because of the high variance of their results (Table 4).

Table 4. Average biodiesel yield, kinematic viscosity, calorific value, density, and cetane number of biodiesel derived from representative palm-based waste cooking oils. For details on abbreviations, refer to Table 1.

Property	FCR	FFR	SP	TR
Biodiesel yield (%)	84.1 ± 5.3 [a]	89.9 ± 2.6	52.6 ± 42.3	93.1 ± 0.2
Kinematic viscosity (mm^2/s)	5.7 ± 1.1	5.9 ± 0.8	28.6 ± 32.6	5.0 ± 0.3
Calorific value (MJ/kg)	39.7 ± 0.2	39.9 ± 0.2	37.7 ± 2.9	39.9 ± 0.1
Density (kg/m^3)	932 ± 7	935 ± 14	962 ± 69	919 ± 9
Cetane number	69.9	70.4	73.3	67.4

[a] Values are the mean $\pm$ standard deviation, except for cetane number, whose standard deviation was between 0.0001 and 0.0126.

The type of restaurant had a significant impact only on the biodiesel yield (p-value = 0.0087). Kinematic viscosity, calorific value, density, and cetane number all showed no significant differences across the restaurant types (p-value = 0.1464, 0.4196, 0.1008, and 0.3634, respectively). This suggests that the yield is more sensitive to cooking conditions than the other properties. Moreover, the average biodiesel yield observed for typical restaurants was close to that expected for virgin oils [34], evidencing that these restaurants would generate the most suitable WCO to produce biodiesel. This is probably due to the less severe cooking conditions employed at these restaurants.

4. Conclusions

Fisher's Least Significant Difference test for multiple comparisons successfully examined the effect of cooking conditions on selected properties of biodiesel produced from palm-based WCO. These oils performed better than other WCO in terms of biodiesel yield regardless of the conditions that they experienced. The yield decreased as the cooking temperature and length of reuse moved upward, whereas the kinematic viscosity was sensitive only to the length of reuse, rising with increasing reuse. Non-compliance with biodiesel standards and technical requirements was observed in some cases. The calorific value did not significantly change unless the cooking conditions were severe. The cetane number dropped as use and reuse decreased, remaining better compared to petrodiesel nonetheless. Typical restaurants would generate the most suitable WCO to produce biodiesel. This is consistent with the less severe cooking conditions employed at these restaurants.

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